

GC/MS: A Practical User's Guide, 2nd Edition

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Reviewing a book is a serious undertaking. To be fair to the author, publisher of book, editor of the journal publishing the review, and those who will read the review, every word must be carefully read. Over the past 15 years, I have reviewed a number of books for the *Journal of the American Society for Mass Spectrometry*, and I take a great deal of care in making sure that I fulfill my obligations to all of these parties. I reviewed the first edition of this book [1] when it was published in 1998 and found it to have very little value; unfortunately, this 2nd edition is no better, perhaps because there is little new material (two additional chapters that do not add anything, an additional appendix of frequently asked questions, and some slight additions or alterations to chapters from the previous edition) and it may be considered to be even worse than the first. The same faults found with the original edition remain. It is not that there is no valuable information in the book, but rather that the valuable information is buried in an overwhelming amount of incorrect information and obscured by a writing style that might be applicable or suitable for people in a small backwoods area of the United States. My criticism of the first edition was corroborated in reviews by other mass spectrometrists [2–4]. However, in the interest of fairness, there were two positive reviews [5, 6] published for the first edition. At least in the case of the *Analytical Chemistry* review [5], it is obvious that the reviewer only looked at the table of contents and did not thoroughly examine the actual text.

Like the first edition, the second edition is divided into three parts: Part I, *A GC/MS Primer*; Part II, *A [sic] GC/MS Optimization*, and Part III, *Specific Applications of GC/MS*. Parts I and II remain essentially unchanged from the first edition with the exception of some minor word changes and very few text additions. The same

confusion between describing packed columns and magnetic-sector instruments as the paradigm for GC/MS in the beginning of Chapter 1 and then changing to open tubular columns and transmission quadrupole mass spectrometers later in the same chapter and in subsequent chapters remains. The same confusing neologisms from the first edition, such as *single-ion chromatogram* (SIC) instead of mass chromatogram, extracted ion chromatogram (EIC), or reconstructed chromatogram (RIC)—any of which would be more appropriate than SIC and *single-ion monitoring* instead of selected ion monitoring for the acronym SIM; *mass fragment spectra* and *mass spectra* to refer to a single mass spectrum (sometimes the author uses *spectra* and sometimes he uses *spectrum* when talking about a single mass spectrum; however, more often than not, he incorrectly uses *spectra* as the singular form); *chemically induced (CI) ionization* instead of chemical ionization (CI) (later in the book he switches to chemical ionization, which is the indexed term, but *chemically induced (CI) ionization* is the term in the glossary); *amu* instead of *m/z*; *molecular mass* regardless of whether a molecular ion, molecule, radical, or fragment ion is being described; and *ansi-CDF* instead of *net-CDF* when referring to standard data formats, remain. The term *turbomechanical pump* is still in the appendix entitled *A Glossary of GC/MS Terms*. In the appendix entitled *GC/MS Selected Reading List*, the number of journals was increased from four to seven; *Analytical Chemistry* is now listed by its correct name instead of *Journal of Analytical Chemistry*; the *Journal of the American Society of Mass Spectroscopy [sic]* no longer heads the list, but has been replaced by the *American Journal of Mass Spectrometry*. The number of listed monographs in this appendix was increased from 6 to 13. The 2nd edition of Jack Watson's *Introduction to Mass Spectrometry*, 1985, is still listed when the 4th edition was published in 2007.

There is an illustration containing a representation of a quadrupole *m/z* analyzer with five rods (Chapter 1, Figure 1.3 *Quadrupole mass spectrometer*); this was also in the first edition and it remains in this edition after being pointed out in one of the reviews of the first edition [4]. Because the author decided to make almost no changes in the first two parts of this book, the section entitled *1.4 Systems and Cost* is exactly the same as it was in the first edition with the exception of stating that prices listed in Table 1.1. Estimated GC/MS Systems Prices is “an educated guess at 2005 system prices” rather than “1997 system prices” as stated in the first edition. However, all the prices for instrumentation in this table are the same for both editions; the descriptions are the same; the cost-saving examples in the text are the same; does anyone suppose that there have been any changes in these areas during the past 10 years? Those only having access to the second edition will erroneously think that

these prices are appropriate for today's instrumentation and not realize they are the same as reported in the first edition.

The compound represented by the mass spectrum in Figure 1.8 *Mass fragment spectra [sic] (mass spectra [sic])* has a labeled abscissa from m/z 30 to m/z 400. The highest m/z value exhibiting a discernible intensity occurs at m/z 111, which appears to be an isotope peak related to the peak at m/z 110. This display wastes a lot of space and would make any attempts at an interpretation difficult at best; however, examination of the spectrum indicates that it does not represent any compound in either the NIST (National Institute of Standards and Technology) or Wiley mass spectra databases. The next peak at a lower m/z value is at m/z 91, which is the base peak, indicating the loss of 19 m/z units from the suspected molecular ion peak. There are labeled peaks at m/z 65 and m/z 51 with relative intensities of about 10%. At first it would appear from these data that the analyte is either benzylfluoride or one of the regioisomers of fluoretoluene. However, examination of the spectra of these four compounds shows that the molecular ion peak at m/z 110 is 60% to 70% of the intensity of the base peak, which is the $[M - 1]^+$ peak. There are no compounds with a matching mass spectrum in either the Wiley or NIST mass spectral databases. There is a possibility that this figure is the mass spectrum of a mixture of two or more compounds. Regardless of what the spectrum represents, it is not good to use such a spectrum displayed the way it is as an example of how mass spectra should appear. This same spectrum appeared in the first edition.

Propagation of errors from the first to the second edition continues in Figure 5.3. It shows a similar compression of a mass spectrum due to the use of a scale of m/z 30 to m/z 400 (highest m/z value peak at 104). This is supposed to be the mass spectrum of 1,3,5,7-cyclooctatetraene; however, the structure displayed with the spectrum contains no double bonds!

Another one of those "what-you-don't-know-can-really-hurt-you" things in this book is the carryover from the first edition of how ions are separated according to their individual m/z values in a transmission quadrupole analyzer. The author does not understand that the amplitudes of the RF and DC are scanned, not the frequencies. The frequency of a transmission quadrupole mass spectrometer's RF generator is fixed by instrument design. This same problem exists in his description of how the 3D quadrupole ion trap functions. The only more bizarre description is found in that of the *Laser Time-of-Flight (GC/TOF-MS GC/MS Systems)*, Section 14.3, in the *Other GC/MS Systems* chapter in Part III. The author starts by describing a GC/TOF-MS system that "uses a 3-kV electron beam to burst ionize the sample from the GC in the mass spectrometer source." He goes on to describe ion detection as one pulse of ions for each m/z value of the designated mass spectral m/z range (maybe back in the days of Wiley and McLaren). He then introduces the concept of time-array detection

saying that by "summing the resulting time windows allows a 10,000-fold increase in sensitivity." This section then goes from the ridiculous to the absurd. It is best to let the book speak for itself:

"The length of the flight tube has historically produced very large, cumbersome TOF instruments. Folding the tube using electrical 'mirrors' to reflect and accelerate the fragment flight stream back down the flight tube to impact the detector has greatly reduced this problem." This paragraph is then followed by:

"Time-of-flight GC/MS systems are rare outside academia. [I am sure companies like LECO, Waters, and JEOL would find this statement very enlightening.] This MS technique is having more success in LC/MS where LC/MALDI-TOF/MS systems are used for the analysis of proteins, peptides, and polynucleotides. (MALDI is short for masser-assisted [sic] laser desorption and ionization.) The liquid stream from the HPLC is mixed with a chromophore, such as cyanocrotonic acid, that will absorb light from the high intensity laser burst in the source. These target dye molecules explode throwing the accompanying protein into the gaseous phase and at the same time chemically ionizing it. Since the free amino groups on the side chains provide multiple ionization sites, a series of multiple charged molecular ions each with a different charge are formed from a single protein. These are repelled down the flight tube and separated at their m/z masses. Analysis of this family of molecular ions, which differ by the size of their charge z , allows calculation of the molecular weight of the original protein. Charges as large as 20–50 on an ion radical allow enzymatic size proteins (MW = 25–60 kDa) to be separated on a TOF mass spectrometer with separating ranges of 0–1000 amu [sic]."

The text regarding TOF instruments in the second edition is unchanged from that in the first edition. Apparently the author is unaware that LECO Corp. (St. Joseph, MI) acquired a TOF GC/MS instrument and its associated technology in 1995 (2 years before the publication of the first edition and 13 years before the publication of the second edition) and has been successfully selling these instruments ever since. They have made advancements in their GC/MS technology, and they have been a leader in Fast GC and 2D GC technology. None of these advancements from LECO or advancements in the areas of Fast GC and 2D GC is even alluded to in this edition of *GC/MS: A Practical User's Guide*. In Chapter 6 *Chromatographic Methods*, there is added information about an Agilent Technologies specific device, the *QuickSwap*. It is implied that this microfluidic device, which creates a constant pressure of make-up gas that purges the column-connection point when the column is not present, may be useful

with other GC/MS systems; but this is not the case. Each manufacturer would need to design a related device specific to their own hardware. In another chapter, the author acknowledges that this device is specific to a single manufacturer (Agilent), but fails to point out that the Varian, Inc., *QuickSwitch* does the same thing.

In Chapter 12 *An Introduction to Structural Interpretation*, there is no mention of accurate mass measurements being used as a basis for determining elemental composition. It is obvious that this chapter is a word-for-word copy from the first edition. The chapter contains a statement, "The previous Wiley database with 225,000 compounds was thought to have up to 8% incorrect structures in it. It is claimed that the current database has been cleaned up and that the structure assignments are more than 98% accurate." However, in Section 7.5 *Data Analysis and Library Searches* in Chapter 7 *Mass Spectrometer Setup and Operation*, the sizes of the NIST (NIST/EPA/NIH) Mass Spectral Database and Wiley Library have been updated. The second edition of *GC/MS: A Practical User's Guide* now reports the size of the NIST05 Database as 190,000 (190,825 actual) spectra of 162,163 compounds (163,198 actual), but who is going to quibble about a few hundred spectra and Wiley Registry, 8th edition (actually the Wiley 8N, which is the Wiley 8th edition [399,389 spectra] with the spectra from the NIST05, the only version of the Wiley databases that Agilent sells) as having 532,573 spectra (actually 532,581 spectra). The author still refers to the NIST Mass Spectral Database as "a database of environmentally significant compounds." The NIST05 Database is a general mass spectral database and not just a database of "environmentally significant compounds". It should also be pointed out that there have never been any attempts by anyone to correct errors in the Wiley Registry. In fact, Fred McLafferty, the erstwhile steward of the Wiley Registry maintained that such errors as those between spectra, structures, and compound names should be retained as they were. On the other hand, the NIST Database has been undergoing evaluations resulting in the replacement and removal of spectra since before 1994 and is the only evaluated general mass spectral database; it does not provide any claims as to the percentage of errors remaining. Unfortunately, the updated spectra numbers in the Wiley Registry did not find their way to Chapter 12.

It is also unfortunate that although several other electron ionization (EI) mass spectral databases are mentioned in Chapter 7, the new boutique EI mass spectral databases published by John Wiley and Sons such as *Mass Spectra of Designer Drugs 2007* and *Mass Spectra of Pharmaceuticals and Agrochemicals 2006*, to name just two, are not cited. Who's the publisher of this book?

Also missing from Chapter 12 is any information about NIST's AMDIS (Automated Mass Spectral Deconvolution and Identification System), which is used for component deconvolution using mass chromatographic profiles as well as the same sort of program provided by

LECO for their proprietary data. There is nothing about the chromatographic peak deconvolution quantitation program from Ion Signature Technology (N. Smithfield, RI). There are also programs from ACD/Labs (Advanced Chemical Development, Inc., Toronto, Ontario, Canada) that would be of interest to GC/MS users. The Cerno Bioscience (Danbury, CT) software routine used to assign accurate mass values to mass spectral peaks acquired using unit-mass resolution instruments such as those from a transmission quadrupole is not mentioned. Again, in all fairness, it must be said that other than the deficiencies and minor problems with the description of the NIST and Wiley mass spectral databases, much of the material in Chapter 12 is relevant and of value; however, the information and word structure of the material in this chapter appear to have been taken directly from one of the four editions of McLafferty's *Interpretation of Mass Spectra*.

Another curious omission from the book is seen in Chapter 8 *Data Processing and Network Interfacing*. The incorrect information in this chapter is far less egregious than that in other chapters. However, in Section 8.5 *File Conversion and Data File Exchange*, there is no mention of *MASSTransit* from Palisade (Newfield, NY) or *GC, GC/MS File Translator Pro* from ChemSW (Fairfield, CA). This section discusses the need for translation of data from minicomputers, which was already dated for the first edition, but certainly could be omitted from this edition. Another example of the author's failure to pay attention to detail is seen in this section with the definition of LIMS as "laboratory information system" as opposed to "laboratory information management system." He did get this one right in the first edition, but somehow had difficulties in this edition.

In Part III *Specific Applications*, there are two new chapters. One of these is Chapter 11 *GC/MS in Forensics, Toxicology, and Space Science*. The less than three-and-a-half pages comprising this chapter are written as a review article. However, like the rest of the book, there are no citations to the literature to describe the various analyses listed under the headings of *Forensic Analyses, Clinical Drug Analyses, Arson and Security Analyses, and Astrochemistry*. No place in the book is there any mention of the use of GC/MS for analyses of flavors and fragrances or the analysis of pesticides associated with commodities. There is a brief mention about "other GC/MS sample analysis methods used for the confirmation of pesticides/PCBs and dioxan/furans" in Chapter 10 *GC/MS in the Environmental Laboratory*, again with no citations, but no real mention of its use for pesticide analyses. This chapter is primarily concerned with U.S. Environmental Protection Agency (EPA) Methods 625 and 624 and cannot really be considered as an encompassing treatment of GC/MS in the environmental laboratory. The material presented on Methods 624 and 625 is correct and has merit, but, of course, it is readily available from many other sources.

The other new chapter in Part III is Chapter 16 *Innovations in GC/MS*. This chapter is comprised of

sections entitled *Microfluidics in GC/MS, Resistance Column Heating, Portable Gas Supply, Portable GC/MS Systems, and New Column Technology*. This, too, is written as a review chapter, but without citations. There is a single reference to a German Web site that has listings and links to various portable and transportable GC/MS systems (<http://www.gcms.de>). This chapter also highlights the author's inability to deal with detail. In talking about various portable instruments, he mentions the *Hapsite* from Indicon [*sic*], Inc., which, of course, should have been Inficon, Inc. The section on new column technology begins with the sentence: "Monolith (probably should be *monolithic*) and micro-WCOT (wall-coated open tubule [I'm sure this should have been *tubular*]) columns are on the *bleeding* edge of new GC column technology." I was uncertain as to whether the author had fallen into one of his folksy forms of rhetoric because GC-column bleed has been the bane of GC/MS or that he actually had meant to say "... on the leading edge of new GC column technology." I did note that *bleeding* was a spell-checker-suggested correction for the misspelling of *leeding*. I think the reason for so many errors of this type is that the author never bothers to go back and read what he has written. That could be the reason that he has had time to publish five books in about 10 years. It is also obvious that no one at the publisher bothers to read the material before it is printed.

Chapter 16 is another missed opportunity to introduce 2D GC and Fast GC, which are becoming important techniques in the field of GC/MS. Another important topic completely ignored is the area of pyrolysis GC/MS. Either in Chapter 16 or at least some place in the book, there should have been a discussion of gas sample valves.

A curious addition to the book in Chapter 13 *Ion Trap GC/MS Systems* is the linear quadrupole ion trap (LIT). The figure shown (source cited as *BioAnalytical Systeme* [*sic*]) is the illustration of the third stage of the Q-Trap instruments from Applied Biosystems, Inc./MDS SCIEX. This is the third stage of a tandem-in-space triple quadrupole instrument used with atmospheric pressure ionization sources associated with LC/MS instruments and it has never been offered as a stand-alone mass spectrometer, not even for LC/MS. There is no mention of the Thermo Scientific *LTQ*, which is the only linear quadrupole ion trap used as a stand-alone mass spectrometer. The author presents the ABI/MDS SCIEX instrument in such a way as to make it appear as a stand-alone instrument. The concept of the ABI/MDS SCIEX instrument does not lend itself to being a stand-alone configuration; and because ABI/MDS SCIEX has no GC/MS products, it is not very likely that they would develop one. Although the author thinks that the LIT might have some potential as a GC/MS system, it would probably be the *LTQ* developed for that purpose; there is no indication that will happen. The main advantage such an instrument would have is in the extension of the upper limit of quantitation, and the author fails to point this out.

Another interesting difference between the first and second edition is the quality of the figures. Many of the figures have significant detail blacked out as if the figures are poor Xerox-type reproductions. The second edition could pass for a cheap copy of the first edition. Many of the figures are provided in a Power Point presentation that is on a CD ROM in a sealed pocket on the inside back cover. Some of these figures are of much better quality than that of the corresponding figure in the text although both the presentation in the text and in the Power Point presentation are of much poorer quality than in the first edition. The Power Point presentation has a number of inappropriate terms such as *chemical impact (CI) source*. This is the third use of the CI abbreviation in this book. The author has also used CI for *chemical ionization* and *chemically induced* in *chemically induced* ionization. All of the figures in the book are not included in the electronic format, and it should be noted that the illustration of the 5-rod quadrupole analyzer is one that is not included. The source of at least two figures is cited in the text; however, like the first edition, the figures copied directly from an early edition of J. Throck Watson's *Introduction to Mass Spectrometry* (Raven, Philadelphia, 1976 1st edition and 1985 2nd edition) are not cited. The CD ROM has individual Microsoft Word files containing the contents of Appendix A: *GC/MS Frequently Asked Questions*, Appendix B: *GC/MS Quick Reference Guide*, Appendix C: *Sources of GC/MS Background Contamination*, Appendix D: *A Glossary of GC/MS Terms*. There is no electronic version of Appendix E: *GC/MS Selected Reading List*. The *Sources of GC/MS Background Contamination* is yet another example of a lack of attention to detail or not bothering to reread or edit a section of the manuscript; overall, this is simply another example of sloppy work. It contains redundancies and erroneous information, although on the whole, it would not be harmful to a GC/MS beginner and it even may be of limited use.

In addition to the improper and inappropriate use of *amu*, the author uses the terms *daughter mode* and *parent mode* when discussing the precursor- and product-ion analysis modes of MS/MS along with a number of other inappropriate or obsolete terms. Some effort has been made to tone down the folksy rhetoric that was so pervasive in the first edition. The statement that a button was pressed *to start the dance* was changed to *start the process*. However, for those not familiar with this 'Missouri backwoods' jargon, it will be difficult to understand many of the passages of this book.

Totally disregarding the glaring omissions, the use of jargon on top of the technically incorrect statements and confusing illustrations make this book potentially more harmful than helpful to a beginner in GC/MS. The book is not without some good information; however, to be able to separate the good information from the incorrect and harmful information will require a level of expertise that the beginner in GC/MS will not possess. The publisher now sells the book by chapters in a PDF format downloadable from the Internet. The entire book is also avail-

able in PDF format. Chapters 10 and 12 are worth having, but maybe not at the individual chapter price.

The author is not totally to blame for this book. The publisher must take some responsibility. It is one thing to commission a product based mainly on consideration of an attractive title and appealing table of contents that would likely sell 2000+ copies, and quite another to painstakingly foster the development of a product that it knows is correct, factual, and will not be harmful to its potential audience. In recent years, publishers have cut back in the area of technical editors in favor of journalism graduates who can be employed for less. This new breed of editor is more concerned with how the table of contents reads or how the book will sell rather than its technical correctness. *GC/MS: A Practical User's Guide* and the other four books that Wiley has published by Marvin C. McMaster since 1994 (first edition of *HPLC: A Practical User's Guide*; the first edition of *GC/MS: A Practical User's Guide*, 1998; *LC/MS: A Practical User's Guide*, August 2005

(reviewed *J. Am. Soc. Mass Spectrom.* **2006**, 17(8), 1193); and the second edition of *HPLC: A Practical User's Guide*, December 2006) are all example of titles that will sell regardless of the quality of the content.

This book reinforces the statement [1] made about the first edition regarding the First Amendment to the United States Constitution and how important it is to the creation and perpetuation of such publications.

References

1. Sparkman, O. D. Book Review: *GC/MS: A Practical User's Guide*. *J. Am. Soc. Mass Spectrom.* **1999**, 10, 364–367.
2. Stein, B. K. Book Review: *GC/MS: A Practical User's Guide*. *Rapid Commun. Mass Spectrom.* **1999**, 13, 755.
3. Westwood, S. Book Review: *GC/MS: A Practical User's Guide*. *Chromatographia* **2000**, 51(9/10), 638.
4. Sanders, J. K. Book Review: *GC/MS: A Practical User's Guide*. *J. Chem. Educ.* **2000**, 77, 1282.
5. Slayton, J. Book Review: *GC/MS: A Practical User's Guide*. *Anal. Chem.* **1999**, 71(13), 412A.
6. Dougherty, R. C. Book Review: *GC/MS: A Practical User's Guide*. *Applied Spectrosc.* **1999**, 53(8), 308A.